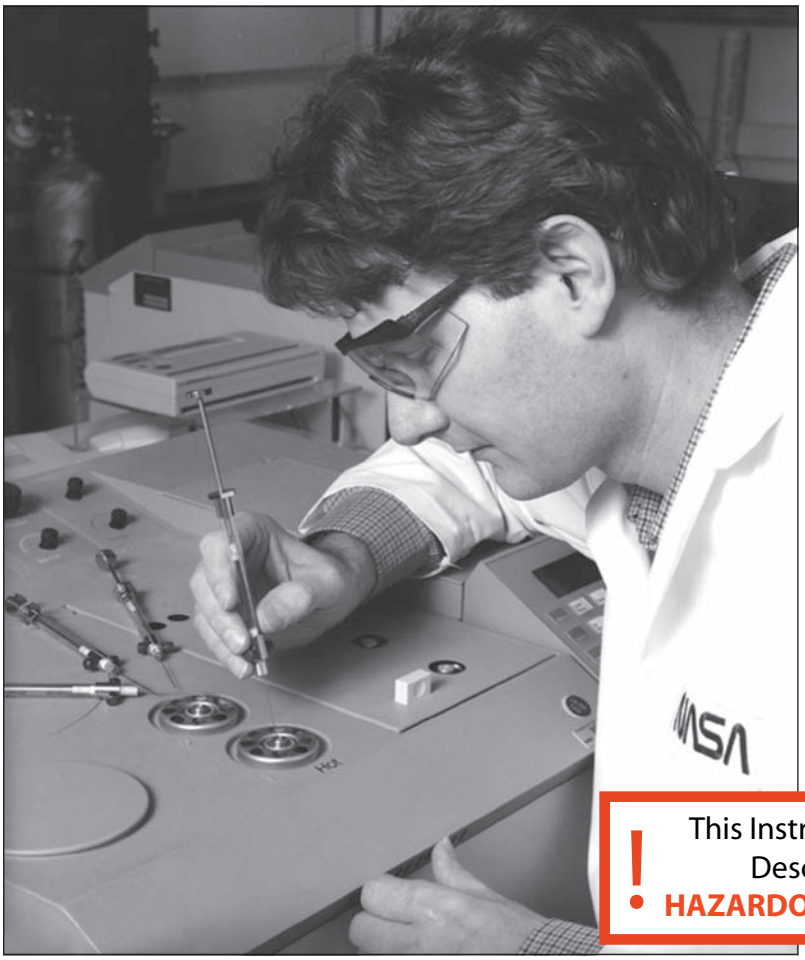
Toxicity Testing

(NASA-STD-6001 Test 7)



! This Instruction Contains Descriptions of **HAZARDOUS OPERATIONS** !

Materials and Processes Laboratory
Materials Test Branch, Building 4623

National Aeronautics and Space Administration
George C. Marshall Space Flight Center
Marshall Space Flight Center, AL 35812

Release Authority	Name	Title	Organization	Date
Office of Primary Responsibility	[s] Gail H. Gordon	Materials Test Branch Chief	EM10	11/15/05
	[s] Dennis Davis	Industrial Safety	QD50	11/16/05

Revision	Date	Originator	Description	Affected Pages
Baseline	2/4/05	Eddie Davis	Document converted from ED36-OWI-039. Previous history retained in system as part of canceled or superseded ISO Document files.	All
A	11/17/05	Eddie Davis	1. Hazardous Operations notification added to cover 2. Section 10 title corrected	Cover, ii 42

This document revises the Organizational Work Instruction (OWI) for the determination of offgassed products per NASA-STD-6001, Test 7, in Building 4623. Any deviation to this procedure shall be approved by the Masterials Test Branch Chief, EM10 and the NASA Toxicity Laboratory manager, via an approved test plan. Any changes to the test shall be noted in the sample test folder or sample log book. It is the responsibility of the Toxicity Laboratory personnel to obtain approval from the NASA Toxicity Laboratory manager for any changes to the test plan.

Any change to this OWI shall be submitted to the approving organizations listed below. The original OWI and all changes shall be maintained by EM10. All documentation must be approved by the appropriate persons and incorporated into the OWI before operation of the modified test can resume.

Concurring organizations:
Building 4623 Test Operations Contractor
EM10 COTR
Environmental Health, AD60M

Contents

This page is deliberately left blank

1.0 Scope

1.1 Scope

The scope of this Organizational Work Instruction (OWI) is NASA-STD-6001, Test 7, Toxicity Testing, as performed in Building 4623 at Marshall Space Flight Center. Testing is performed using analytical methods, including gas chromatography and mass spectrometry.

1.2 Purpose

The purpose of Test 7 is to determine the identity and quantity of volatile off-gassed products from materials and flight hardware. The test attempts to simulate the most extreme conditions that astronauts can tolerate in a spacecraft interior without causing bodily harm. After testing, materials are rated for use, and flight hardware items are given a pass or fail status.

1.3 Applicability

This instruction applies to the Chemistry Team, Materials Test Branch, of the Materials and Processes Laboratory

2.0 Applicable Documents

Agilent 6890 Series Gas Chromatograph Operating Manual, Volume 1 - General Information. Agilent Technologies, Inc. January 2000.

Agilent ChemStation. Understanding Your ChemStations. Agilent Technologies, Inc. 2001.

EM10-OWI-CHM-042. *Test Sample Preparation for Testing in Building 4623.*

EM10-OWI-CHM-050. *Building 4623 Guidelines for Test Operations.*

EM10-OWI-CHM-051. *Receipt, Handling, Prioritization, and Data Requirements for Samples Submitted for Testing in Building 4623 of the Materials and Processes Laboratory.*

EM10-OWI-CHM-058. Chemical Handling Plan for Building 4623.

Envirolink GC/LC-MS Users' Manual. ProLab Resources. 1994.

Finnigan INCOS Series GC/MS Systems Getting Started Manual. Finnigan Mat. 1992.

Finnigan INCOS XL Series Systems Operator's Manual (Vols. 1 & 2). Finnigan Mat. 1992.

Finnigan Trace MS (Including Voyager and MD Series) Hardware Manual. Revision B. Thermoquest. 1999.

Finnigan Trace MS (Including Voyager and MD Series) Getting Started. Revision B. Thermoquest. 1999.

Instruction Manual. GC8000 Top Series. Revision A2. CE Instruments. November 1996.

MPD 1840.3. *MSFC Respiratory Protection Program.*

MPR 1040.3. *MSFC Emergency Plan*.

MPR 1840.2. *MSFC Hazard Communications Program*.

MPR 8715.1. *MSFC Safety, Health, and Environmental (SHE) Program*.

MSFC-RQMT-2918. *Requirements for Electrostatic Discharge Control.*

MWI 3410.1. *Personnel Certification Program*.

MWI 8621.1. *Close Call and Mishap Reporting and Investigation Program*.

NASA-STD-6001. *Flammability, Odor, Offgassing and Compatibility Requirements and Test Procedures for Materials in Environments that Support Combustion.*.

Nicolet Gas Cell User's Guide. Nicolet Instrument Corp. 1997.

Nicolet Nexus 670 User's Guide. Nicolet Instrument Corp. 1997.

Perkin-Elmer Autosystem GC/FID Operator's Manual.

Perkin-Elmer Operator's Manual Autosystem GC. 1993.

PE Nelson Model 1022 Personal Integrator Installation and Setup Guide. August 1993.

Tekmar 3100 Purge and Trap Concentrator Users' Manual. Tekmar-Dohrmann. 1998-1999

Tekmar 6000 Aerotrap Desorber Concentrator Users' Manual. Tekmar-Dohrmann. 1995.

Xcalibur. Getting Productive: Creating and Searching Libraries. Revision A. Thermoquest. 1999.

Xcalibur. Getting Productive: Qualitative Analysis. Revision B. Thermoquest. 1999.

Note: Personnel **shall** always **refer** to the current version of each applicable document.

3.0 Definitions

3.1 Definitions

Assembled article. Any component or assembly of components that is not a single material.

Materials and Processes Technical Information System (MAPTIS). The database used by NASA to evaluate materials and flight hardware.

Non-flight assembly. Any assembly not certified as flight hardware, such as backup assemblies to the flight hardware or items tested solely for qualification.

Offgassed constituent. An organic or inorganic constituent evolved as a gas from a material or assembled article.

Offgassing. The evolution of gaseous products from a liquid or solid material into an atmosphere.

Post-load CO/CH_4 blank. Analysis of the test atmosphere in the sample container after the test article has been loaded and purged with high-purity air but before the 72-hr test. Any CO or CH_4 detected from this analysis shall be subtracted from the final results.

Sample container. Aluminum and stainless-steel chambers used to enclose and seal materials and hardware for the toxicity test. (Section 9.1, Table 2 contains a list of chambers and their volumes available for use at MSFC.)

Spacecraft Maximum Allowable Concentration (SMAC.) The maximum concentration of an offgassed product that is allowed in the habitable area of a spacecraft for a specified flight duration.

Test 7. The determination of offgassed products in accordance with NASA-STD-6001.

Toxic hazard index (T). The value determined by calculating the ratio of the projected concentration of each offgassed product to its SMAC value and summing the ratios for all offgassed products without separation into toxicological categories.

Trace constituent. An offgassed product detected and identified but not at quantifiable levels.

Unidentified constituent. Any constituent that is detected on the mass spectrometer and/or flame ionization detector where no functional group can be isolated. A SMAC value of 0.1 is assigned to these constituents.

3.2 Acronyms

CO	Carbon monoxide
CH_4	Methane
FID	Flame Ionization Detector
GC	Gas Chromatography
GC/MS	Gas Chromatography/Mass Spectrometry
LN_2	Liquid nitrogen
MAPTIS	Materials and Processes Technical Information System
MLU	Max Limit Units
MLW	Maximum Limit Weight
MSDS	Materials Safety Data Sheet
SMAC	Spacecraft Maximum Allowable Concentration

4.0 Instructions

All operations of Toxicity Laboratory equipment **shall be conducted** using the applicable documents referenced above (section 2). All critical measuring devices, *e.g.*, chart recorders, balances, ovens, aerostats, **shall** be in current calibration (section 9.3).

4.1 Pre-Test Conditions

The following conditions **shall be met** before performing Test 7:

4.1.1. Testing of flight hardware assemblies is scheduled through Toxicity Laboratory personnel.

4.1.2. A program representative accompanies the flight hardware to the laboratory and is present during loading and unloading.

4.1.3. ESD-certified Toxicity Laboratory personnel are present for loading and unloading of flight hardware to ensure Quality Assurance and Safety. [Personnel **shall refer** to MSFC memo EH42B (97-319).] A Pre-/Post-Test Certification Sheet (form EM10-F-CHM-032) shall be completed for flight hardware items. (Section 7.3, Figure 4 shows a typical certification sheet.)

4.1.4. The test requester has completed an MSFC test request worksheet for each individual toxicity test. (Section 7.3, Figure 5 illustrates the test request worksheet.)

4.1.5. All off-the-shelf items and materials have a product data sheet from the manufacturer. Materials for testing have an MSDS sheet.

4.1.6. All non-flight assemblies for testing have a materials list provided.

4.1.7. The test requester has compiled and submitted the above information, along with the material or non-flight assembly, to:

Bldg. 4623
Attn.: Sample Coordinator EM10
MSFC, AL 35812

4.1.8. The sample coordinator (EM10) creates an MSFC Test Request (EM10-F-CHM-002) in the Materials and Processes Technical Information System (MAPTIS).

4.1.9. The non-flight assembly or material **shall be assigned** an appropriate test number and is accompanied by a folder containing all pertinent information.

4.1.10. Part number and serial number are verified for each assembly. Personnel **shall ensure** these numbers are recorded in the Toxicity Testing Log Book.

4.1.11. The test requester has provided dimensions for assemblies to ensure proper fit and availability of the sample container.

4.1.12. Personnel **shall verify** all necessary pre-test items before sample loading.

4.2 Pre-Test Procedures for Sample Containers

Toxicity personnel **shall perform** the following procedures:

4.2.1. At the conclusion of a toxicity test, **remove** the material or assembly from the sample container. **Place** the open sample container in the oven at 49 (±3) °C for 72 hours to drive off any contamination products from the previous test.

4.2.2. After the 72 hours, **remove** the sample container from the oven. **Bolt** the lid on the container, and **seal** the container using a tin/lead solder gasket to ensure a leak-tight seal.

4.2.3. **Open** the sample container valves, and **purge** the container with high- purity air until a minimum of five atmosphere exchanges have been accomplished. **Allow** the atmosphere within the sample container to equilibrate. **Close** the valves, and **replace** the sample container in the test oven for the thermal conditioning portion of the blanking procedure. The thermal conditioning is performed for 72 hours at 49 (±3) °C.

4.2.4. After this 72-hour period, **remove** the sample container from the oven, and **allow** it to cool to ambient temperature.

4.2.5. **Analyze** the atmosphere of the sample container to verify cleanliness by using gas chromatography methods. (**Refer** to *Autosystem GC/FID SOP* and *Perkin Elmer Operator's Manual Autosystem GC.) If any contamination products are detected*, **repeat** the blanking procedure (steps 4.2.1-4.2.4 above), and **reanalyze** the sample container. In the unlikely event that the blanking procedure can not be repeated, **subtract** any contaminants from the offgassed results. Certification of cleanliness is based on MSFC testing procedures.

4.2.6. **Retain** the chromatogram of the blanking procedure as a record. Each sample container has a corresponding folder that is stored in the Toxicity Laboratory file cabinet (Room 100). This chromatogram is then included in the sample test folder for the next toxicity test performed in that specific sample container.

4.2.7. After the blanking procedure is completed, **leave** the sample container in the sealed configuration until the next sample is to be loaded.

4.3 Sample Preparation

Toxicity personnel **shall perform** the following procedures:

Refer to EM10-OWI-CHM-042, *Test Sample Preparation for Testing in Building 4623*, for information on standard toxicity sample preparation. NASA-STD-6001 contains additional sample preparation information.

4.3.1. Test material. The desired ratio of test material to sample container volume is 5 (±0.25) g/liter. The 4.23-liter chambers are most commonly used; therefore, a 21.2-g sample size meets the requirement for materials testing in these chambers.

4.3.2. Assemblies. The desired ratio of assemblies to sample container volumes is approximately 1:3.

Note: Assemblies typically require no sample preparation.

4.3.3. Materials on a substrate. For materials that require application to a substrate, *i.e.*, coatings and adhesives, **use** a 4 in. x 7.5 in. x 0.003 in. aluminum substrate. **Apply** the material to the substrate according to the manufacturer's specification until the proper sample weight is obtained.

4.4 Pre-Test Photography

The *sample preparation technician* **shall take** a pre-test photograph of all materials and assemblies and **shall place** three photograph in the sample test folder. *If the test sample photos are not in the folder*, then *Toxicity Laboratory personnel* **shall ensure** that the photographs are made. *Toxicity Laboratory personnel* **shall take** all Flight Hardware photographs.

4.5 System Setup and Sample Loading

Toxicity personnel **shall perform** the following procedures:

4.5.1. After the Sample Preparation Laboratory has generated the sample test folder, assigned an item number, and sealed the material in polyethylene bags, the Toxicity Laboratory receives the sample and folder. **Store** the sample in the Toxicity Laboratory sample cabinet (Room 102) before testing.

4.5.2. **Schedule** testing according to the following priorities:

Test Category/Sample Priority	Completion Date
Flight Hardware	2 weeks (or sooner)
Priority 0 Samples	1 week
Priority 1 Samples	2 weeks
Priority 2 Samples	3 weeks
Priority 3 Samples	4 weeks
Priority 4 Samples	1 year

Note: The NASA Toxicity Laboratory Manager shall approve all final test scheduling.

4.5.3. Before loading the sample, **enter** all pertinent data associated with a sample in the *Toxicity Testing Log Book*. (Section 7.3, Figure 6 shows a sample data sheet from the log book.)

4.5.4. On the specified load date, **unbolt** the sample container lid. **Remove** the sample from the storage container, and **transfer** it to the sample container.

4.5.5. *If the assembly or flight hardware is certified as ESD sensitive,* **perform** loading procedures at the certified ESD station in accordance with MSFC RQMT 2918. **Wear** static-sensitive gloves to load samples into the sample container.

4.5.6. After the sample is loaded, **bolt on** the top, and **seal** the sample container with a tin/lead solder gasket to ensure a leak-tight seal.

4.6 Detailed Test Procedure

Toxicity personnel **shall perform** the following procedures:

4.6.1. **Open** the sample container valve and port, and **purge** the system with high-purity air. **Remove** the purge line, **allow** the atmosphere within the sample container to equilibrate, and **close** the valve and port.

4.6.2. **Verify** the gas cylinders that supply the Gas Chromatographs (GCs) and other primary Toxicity Laboratory instruments have sufficient pressure (80 psi) and that the carrier flows are adequate for analysis.

4.6.3. **Analyze** the test atmosphere using GC/FID methods by performing a post-load carbon monoxide/methane (CO/CH_4) blank run (section 9.1, Figure 12). **Subtract** the amounts present in this blank from the final offgassed amounts.

4.6.4. **Place** the sealed sample container in the laboratory oven, which is constantly operating at 49 °C.

4.6.5. **Thermally condition** the sample in a laboratory oven at 49 (±3) °C for 72 (±1) hours. Any deviation from these conditions shall be approved by EM01 management. **Record** any anomalies on the Pre-/Post-Test Certification Sheet, an example of which appears in Figure 4.

4.6.6. After thermal conditioning, **remove** the sample container from the laboratory oven. **Refer** to section 9.3, Calibration, steps 9.3.3.2 through 9.3.3.10, for the procedure for data acquisition. **Print** the entire chromatogram at the end of a sample run.

Note: Use gas-tight syringes for removal of gases from all sample containers and injection of gases into the instruments.

4.6.7. **Withdraw** a gas sample from the sample container. **Inject** the gas sample into the Tekmar 3100 purge and trap (Figure 1) for concentration and desorption. The gas sample is desorbed from the Tekmar 3100 to the Finnigan INCOS XL GC/MS (section 9.1, Figure 13), which is the primary instrument for ***identification*** of the offgassed constituents. **Label** and **identify** all peaks detected on the Finnigan by chemical name or compound functional group. (**Refer** to the *Finnigan INCOS XL GC/MS SOP* and *INCOS XL Series Systems Operator's Manual*, *Vols. 1 & 2*.)

Figure 1. Injection of Gas Sample into the Tekmar Instrument.

4.6.8. **Inject** an additional gas sample into the Tekmar Aerotrap 6000 Purge and Trap for concentration and desorption analyses. The gas sample is desorbed from the Tekmar 6000 onto the Finnigan Voyager GC/MS. The Voyager GC/MS is also used to identify offgassed constituents. The data obtained are used to support the INCOS 50 GC/MS data.

4.6.9. **Allow** the sample chamber to cool to ambient temperature. (**Use** a digital thermometer and thermocouple to verify ambient temperature of the sample container.)

4.6.10. **Withdraw** a minimum of two additional samples from the sample container. **Directly inject** these samples into the Autosystem GC/FID (capillary column) (section 9.1, Figure 11), which is the primary instrument for ***quantification*** of the offgassed constituents. (**Refer** to section 9.3.4 and the *Perkin Elmer Operator's Manual Autosystem GC*.) This GC is equipped with two identical columns that use the same temperature program as does the Finnigan GC/MS. The instrument produces integrated area counts.

4.6.11. **Withdraw** two additional samples from the sample container. **Directly inject** these samples into the Agilent 6890 (packed column) (section 9.1, Figure 12), which determines the CO and CH_4 concentrations. (**Refer** to section 9.3.5 and the *Agilent 6890 Series Gas Chromatograph Operating Manual*.) This instrument is equipped with a molecular sieve column that is designed with the use of a specialized catalyst. The instrument produces integrated area counts.

4.6.12. Each instrument produces a gas chromatogram. **Keep** this chromatogram in the sample test folder.

Note: Peaks identified on the Finnigan GC/MS systems are based on premeasured retention times, fragmentation patterns, and library match identification, using four separate libraries. Two of these libraries were received with the instrument as software packages, and two were built using available in-house standards. Current hardcopies of the in-house libraries are kept in the Toxicity Laboratory.

4.6.13. **Identify** the chromatographic peaks detected on the Autosystem GC/FID by matching them with corresponding peaks from the Finnigan GC/MS analysis (Figure 2). **Consider** all peaks detected from the Autosystem analysis as *quantifiable*, peaks identified on the Finnigan only as *trace constituents*, and any constituent detected on the mass spectrometer and/or flame ionization detector where no functional group can be isolated as an *unidentified constituent*. A SMAC value of 0.1 is assigned to these unidentified constituents.

4.6.14. The sample shall be analyzed on the Nicolet Fourier Transform Infrared (FTIR) instrument. **Follow** the procedure for calibration of the FTIR (section 9.3.6). **Validate** that the calibration is within the specified limits, and **pull** a vacuum on the gas cell. **Transfer** the headspace gas from the sample chamber to the infrared gas cell. **Analyze** the sample by selecting **Collect Sample**. **Review** the sample data for formaldehyde (peaks at 1745 and 2896 cm^{-1}**)** and ammonia

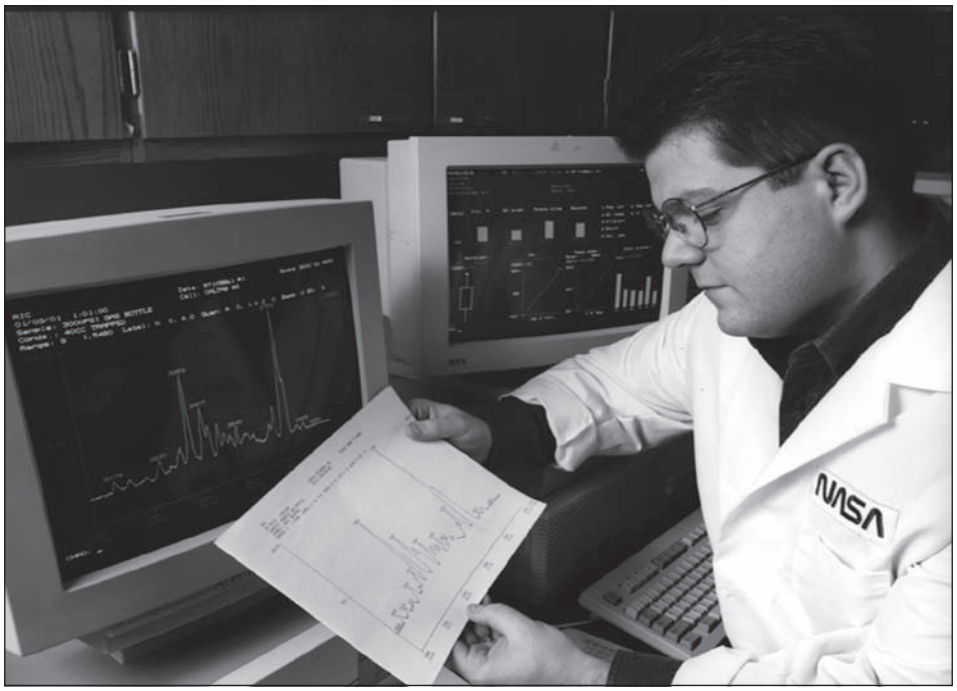


Figure 2.
Comparison of Chromatographic Peaks.

(931 and 966 cm^{-1}**). Review** the data for any other unusual peaks. **Save** and **print** the sample data.

4.6.15. **Enter** the gas code and integrated area counts for each offgassed constituent into the MAPTIS database. MAPTIS then determines the concentration of each offgassed constituent, using the average area count and a relative flame response factor. Concentrations are given in micrograms per gram for materials and in total micrograms for assemblies.

4.6.16. After all analyses are complete, **check** the sample test folder to verify that all the following supporting data are included:

- Pre-/Post-Test Certification Sheet (flight hardware only), form EM10-F-CHM-032 (section 7.3, Figure 4)
- Sample preparation data sheet (materials) (section 7.3, Figure 10)
- Test request, form EM10-F-CHM-002, current revision (section 7.3, Figure 5)
- Copy of completed log sheet, form EM10-F-CHM-033 (section 7.3, Figure 6**)**
- CO and CH_4 blank chromatogram
- Sample container Autosystem GC blank chromatogram
- Autosystem GC chromatograms from sample analysis
- Finnigan INCOS XL chromatogram
- Finnigan Voyager chromatogram
- Autosystem calibration standard chromatogram
- CO/CH_4 calibration standard chromatogram
- Nicolet Nexus 670 FTIR standard spectra
- Nicolet Nexus 670 FTIR sample spectra
- Additional supporting data, *e.g.,* mass fragment patterns, structures

- Equipment calibration sheet (section 7.3, Figure 7)
- MAPTIS rating sheets (section 7.3, Figures 8 and 9)
- MSDS (materials)
- Previous MAPTIS test data (if available)
- Photographs
- Post-load CO/CH_4 blank
- MAPTIS information sheets
- Cure sheets, if applicable.

4.6.17. After the Toxicity Laboratory personnel and the data analyst have reviewed the data, the NASA Toxicity Laboratory Manager reviews the sample test folder for each test. Flight hardware tested receives either an ***A*** for passing or an ***X*** for non-passing status. In the event that the test results yield an ***X*** rating, the requester is notified and given the option to schedule an immediate retest.

4.6.18. The NASA Toxicity Laboratory manager generates a memo that contains the final test report for each sample tested. The memo (section 7.2, Figure 3) shall be reviewed and signed by EM01 management.

4.6.19. EM10 personnel forward the signed memo and final test report to the test requester.

4.7 Data Recording and Reduction; Post-Test Photography

4.7.1. Data Recording and Reduction

4.7.1.1. The MAPTIS database performs the calculations for each test, based on the data entered by Toxicity Laboratory personnel. The data include:

- Item number
- Material code
- Test date
- Sample weight
- Sample size
- Sample container size
- Sample container free volume
- Sample cure information
- Gas codes and names of each reported offgassed constituent, including integrated area counts.

Note: Trace constituents shall be reported but not used in the calculation of the toxic hazard index (T-100 or T Value).

4.7.1.2. Assemblies. The offgassed products included in the final report for assemblies and flight hardware are reported in total micrograms for each constituent detected. The final report shall indicate a pass or fail status. As-

semblies and flight hardware pass with a summation of a T Value of 0.5 or less. Assembly and flight hardware reports shall include:

- The summation of T (T Value)
- Maximum Limit Units (MLUs)
- Assembly rating. (Section 7.3, Figure 8 contains a sample toxicity rating sheet for assemblies used in the Shuttle, Spacelab, and SS Airlock.)

4.7.1.3. Materials. The offgassed products included in the final report for materials are reported in micrograms/gram of each constituent detected. The final report rates each material for spacecraft use. (**Refer** to section 7.1.3.1 for materials ratings.) The rating given to each material corresponds to a Maximum Limit Weight (MLW). Material reports shall include:

- The T Value
- MLW
- Materials rating. (Section 7.3, Figure 9, contains a sample toxicity rating sheet for materials used in the ISS Module.)

Note: The T Value is calculated for each offgassed constituent based on offgassed amounts, spacecraft volume, and the SMAC value for each individual constituent.

Note: Section 7.1 contains Toxicity Calculations and Ratings.

4.7.2. Post-test photographs are only required when a noticeable change occurs in the appearance of a test sample during analysis. *Toxicity Laboratory personnel* **shall photograph** such samples after analysis. Copies of the post-test photographs shall be placed in the test folder. Photographs shall be retained indefinitely.

5.0 Notes

Custodians for EM10-OWI-CHM-039	
Master List and Document Control	EM10 Management Support Assistant
Alternate Document Control	EM10 Group ISO Representative
Records	Materials Test Bramch ISO Representative
Calibration	Materials Test Bramch Calibration Contact
Memoranda	Materials Test Bramch ISO Representative

6.0 Safety Precautions and Warning Notes

6.1 Hazards

The nature of Test 7, Toxic Offgassing, involves several potential hazards. These include:

Warning
Death, severe personal injury, or loss of major equipment may result if maintenance or operating procedures, techniques, restrictions, etc., are not followed exactly.

- Transporting compressed gas cylinders
- Working with cryogenic liquids
- Lifting and moving sample containers
- Loading and unloading flight hardware
- Handling of hazardous chemicals
- Handling gas tight syringes.

6.2 Safety Precautions

Toxicity Laboratory personnel **shall**:

6.2.1. **Observe** normal laboratory safety rules.

6.2.2. **Plan** testing so that two Toxicity Laboratory chemists are in the test area or that one Toxicity Laboratory chemist is in the test area one other person is in Building 4623 during testing. (Thermal conditioning periods may be unattended.) For specific personnel requirements, refer to section 4.7, Personnel Control (Facility Management), of EM10-OWI-CHM-050, *Building 4623 Guidelines for Test Operations*.

6.2.3. **Wear** safety apparel appropriate for the test specimens and conditions**.** In accordance with OSHA requirements, **read** the Materials Safety Data Sheets (MSDSs) for all chemicals used or encountered during testing. **Read the test material's MSDS to ensure familiarity with all safety precautions associated with the material. Verify that all Toxicity Laboratory personnel are aware of all highly hazardous, reactive, or toxic components of the test material.**

6.2.4. When operating Toxicity Laboratory equipment, **refer** to the safety section of each operator's manual, located in the Toxicity Laboratory.

6.2.5. **Smoking is not permitted** in Building 4623. The test area is generally an oxygen-enriched environment. Open flames or other high-temperature sources are not permissible in the testing area while enriched-oxygen conditions exist.

6.2.6. While testing materials or assembled articles for Test 7, **avoid inhalation** of any vapors produced by the test. **Pay particular attention** during removal

of samples from the sample container. **Perform** the unloading and removal of the material or assembled article in a well-ventilated area, using a fume hood if necessary.

6.2.7. **Verify** that no lights or sirens are on outside the Toxicity Laboratory before entering to ensure that the oxygen level indicated by the gauge reads between 19.5 and 23.5% oxygen.

6.2.8 When handling cylinders and dewars or when making connections for compressed gases and/or liquids, **refer** to *Working Safely with Compressed Gases and Cryogens* and *NSTC 313-Cryogenics Safety.* (**See** the test engineer for these resources.) **Comply** with the suggestions inside these presentations.

6.3 Special Precautions Associated with Compressed Gases and Liquids

6.3.1. All operations involving compressed gases and liquids shall be conducted using the buddy system.

6.3.2. All operating personnel shall be instructed on the nature of hazards associated with compressed gases and liquids.

6.3.3. Before removal of any component of the system for servicing, the operator shall secure and inspect the system to ensure that no unsafe condition exists.

6.3.4 Personnel shall perform continuous monitoring, *e.g.,* check operating pressures, look for leaks, listen for unusual noises, during all operations. Personnel shall ensure that oxygen leak levels are adequate throughout operations.

6.4 Emergency Shutdown Procedure

Not applicable; Toxicity Laboratory equipment does not have to be shutdown to be considered safe in an emergency.

6.5 Accident Reporting

6.5.1. From a safe location, the *test operator* **shall immediately call 911** and **shall notify** the EM10 Materials Test Branch Chief or the Toxicity Laboratory manager.

6.5.2. From a safe location, the notified *EM10 Materials Test Branch Chief/Toxicity Laboratory manager* **shall immediately report** the accident to the NASA Safety Monitor and the appropriate supervisor(s).

6.6 Emergency Response Plan

Emergency procedures and plans for Building 4623 are incorporated into the OWIs and are stated in MPG 1040.3H, *MSFC Emergency Plan*. Plans **shall** be modified if operations change in a significant manner.

6.7 Mishap Reporting

"Each employee is responsible for reporting emergencies, unsafe or potentially unsafe conditions, mishaps and close calls in the workplace.[1]"

Toxicity Laboratory personnel shall:

Report all mishaps in the Toxicity Laboratory in Building 4623 to the *Toxicity Laboratory manager*, who shall **report** the mishap to the *area coordinator/Safety Monitor*, who shall **report** the mishap or close call in accordance with MWI 8621.1, *Close Call and Mishap/Incident Reporting and Investigation Program*.

- For all Type A & B mishaps, the *area coordinator/Safety Monitor* shall immediately (as soon as possible) **initiate** an initial verbal report to the Center Director and S&MA Director.

  Note for contractor employees: In the absence of the Toxicity Laboratory manager and the area coordinator/Safety Monitor or other NASA employee, any employee is authorized to initiate verbal notification of the Center Director and S&MA Director immediately (as soon as possible).

- For all mishaps and close calls, flash report is required and shall be generated within 4 hours of the mishap occurrence. The *employee reporting the mishap or close call* **shall notify** his/her supervisor immediately. The *employee's immediate supervisor* **shall call** 544-4357, Option 0, to generate the flash report. In addition, the *employee's immediate supervisor* **shall submit** NASA Form 1627 to S&MA within 6 calendar days. All mishaps **shall** be reported in accordance with MWI 8621.1, *Close Call and Mishap/Incident Reporting and Investigation Program.*

Mishaps or close calls that occur while performing Toxicity Laboratory analyses in facilities other than Building 4623 shall be reported to that facility's building manager or test engineer responsible for developing that facility's emergency procedures, in accordance with MWI 8621.1, *Close Call and Mishap/Incident Reporting and Investigation Program.* The *employee reporting the mishap or close call* **shall also notify** his/her supervisor immediately. The *employee's immediate supervisor* **shall call** 544-4357, Option 0, to generate the flash report. The *employee's immediate supervisor* **shall submit** NASA Form 1627 to S&MA within 6 calendar days. All mishaps **shall** be reported in accordance with MWI 8621.1.

[1] MWI 8621.1 *Close Call and Mishap/Incident Reporting and Investigation Program.* March 27, 2000. pg. 7.

7.0 Attachments, Data, Reports, and Forms

7.1 Attachments

Unless otherwise noted, all data are normalized to a 7-day (168-hour) duration and a volume of 65 cubic meters for Shuttle and 118 cubic meters for Space Station. Materials/Assemblies are rated for use, based on analysis performed per NASA-STD-6001.

7.1.1. Calculation for Materials

7.1.1.1. Definitions of Terms

- TX1 = Offgassed amount for each constituent in micrograms per gram of material
- TX2 = Offgassed amount for each constituent in milligrams per cubic meter.
- Spacecraft Maximum Allowable Concentration (SMAC) = maximum concentration of an offgassed product that is allowable in the habitable area of the spacecraft for a specific duration. SMAC values for each offgassed constituent are reported in milligrams per cubic meter (JSC limit).
- TC = T value for each constituent (dimensionless)
- T100 = Total T value for 100 lb of material (The addition of all T values for the entire test is the summation of T.)
- MLW= Max Limit Weight in pounds of material

7.1.1.2. Calculation of TX1: total micrograms of offgassed constituent/sample weight in grams.

7.1.1.3. Calculation of TX2: Convert micrograms/gram to mg/m^3, using the conversion factors: Shuttle: TX2 = TX1 x 0.6978; Space Station: TX2 = TX1 x 0.3845.

7.1.1.4. Calculation of TC: TX2/SMAC. Calculate TC for each constituent.

7.1.1.5. Calculation of MLW: 0.5 / T100 x 100 lb.

Note: Trace constituents are indicated on final reports; however, these are not used in the calculation of the T100 value.

Note: All Flight Hardware/Assemblies will be tested in an as-used configuration.

7.1.2. Calculation for Assemblies. The MAPTIS database performs the following calculations.

7.1.2.1. **Convert** total micrograms for each individual constituent (taken from the test report) to total milligrams by dividing by 1000.

7.1.2.2. **Divide** the total milligrams by the spacecraft volume. (Use 65 m^3 for Shuttle and 118 m^3 for Space Station.) Units will be mg/m^3.

7.1.2.3. **Divide** the results obtained in step 7.1.2.2. by the constituent's SMAC value. (SMAC units are also mg/m^3.) This gives the dimensionless T value for that specific constituent.

7.1.2.4. **Add** together the T values (TC) for the summation of T.

7.1.2.5. **Calculate** Maximum Limit Units (MLUs): 0.5/Sum T.

7.1.3. Ratings. Ratings are based on the MLW.

7.1.3.1. Materials

K = MLW of 100 lb or greater
H = MLW of 50 to 99.999 lb
A = MLW of 10 to 49.999 lb
V = MLW of 5 to 9.999 lb
X = MLW of 0 to 4.999 lb.

7.1.3.2. Assemblies

A = Summation of T equal to or less than 0.5.
X = Summation of T greater than 0.5.

7.2 Reports

Figure 3 presents the memo format for EM10 test reporting.

7.3 Forms

Figures 4 through 10 are representations of the forms required for conducting Test 7, Toxicity Testing.

EM10 (Year-Number)

(Date:)

TO: Organization/Supervisor

FROM: EM10/Gail Gordon
SUBJECT: Sample and/or Test Procedure Conducted

The subject samples have been analyzed as requested. (Describe the test procedure conducted, along with the results of the procedure and any other pertinent information.)

Gail Gordon
Materials Test Branch Chief
Materials and Processes Laboratory

cc:
Test Requester
Test Conductor
Other Appropriate Personnel

Figure 3.
Typical EM10 Test Report Format.

Figure 4.
Typical Pre-/Post-Test Certification Sheet (used for flight hardware only).

Pre-/Post-Test Certification Sheet

Date: _____ EM10 Tox Lab Number: _____

Hardware Description: _____

_____

Serial Number: _____ Part Number: _____

Category 1 Test Instrumentation: Chart Recorder: _____
Balance: _____

Category 1 Calibration Gases: _____

Pre-Test Certification

Contractor/User Representative (C/UR) Name (print): _____

	EM10	C/UR
1. Hardware visibly clean?	Y ☐ N ☐	Y ☐ N ☐
2. Test configuration cleaned? ready for test? (test chamber, oven, test procedure, personnel)	Y ☐ N ☐	Y ☐ N ☐
Comments/concerns: _____		
3. (If applicable) Contractor handling procedures ready for test?	Y ☐ N ☐	Y ☐ N ☐
4. Test procedure ready for test?	Y ☐ N ☐	
5. Test facility monitoring schedule developed?	Y ☐ N ☐	

Test

1. Test procedure modified during test?	Y ☐ N ☐	
Explain all modifications: _____		
2. Did any test anomalies occur?	Y ☐ N ☐	Y ☐ N ☐
Explain all anomalies: _____		

Post-Test Certification

C/UR Name (print): _____

1. Article ready for removal from test chamber?	Y ☐ N ☐	Y ☐ N ☐
2. Article undamaged and ready for packaging?	Y ☐ N ☐	Y ☐ N ☐
3. (If applicable) Handling procedures completed?	Y ☐ N ☐	Y ☐ N ☐
Modifications to handling procedure approved?	Y ☐ N ☐	Y ☐ N ☐

1/05 EM10-F-CHM-032

Note: Representative Data Sheet. Refer to Forms Master list for current version.

NASA / MSFC

Materials Combustion Research Facility Test Request Worksheet

Requester Information:

Name: | Phone:
Organization/Employer: | Fax:
Street Address: | E-Mail:
City:
State: Zip Code: Country:
Request Date: | Requester's Tracking Number (if any):

Material/Assembly Information

Material/Assembly Name: | Part Number: / Serial Number:
Material Batch/Lot #: | Application:
Material Specification: | Project:
Cleaning Specification/Method:

Vehicle:

☐ Space Station ☐ Shuttle Cargo Bay ☐ SLI
☐ Shuttle Crew/Experiment Area ☐ Unmanned Mission ☐ Other (list):

Manufacturer's Information	***Supplier's Information***
Name:	Name:
Street Address:	Street Address:
City:	City:
State: Zip Code: Country:	State: Zip Code: Country:
Phone: Web Site:	Phone: Web Site:

Cure Information

Is cure required? Yes ☐ No ☐ | Has requester performed cure? Yes ☐ No ☐ | Is MCRF to perform cure? Yes ☐ No ☐

Provide cure method, whether performed by requester or to be performed by MCRF, in the following table:

Step	Time *(hr)*	Mix Ratio *(if applicable)*	Temperature *(°F)*	Pressure *(psia)*
1.		By Wt ☐ or Vol ☐ ?		
2.		By Wt ☐ or Vol ☐ ?		
3.		By Wt ☐ or Vol ☐ ?		
4.		By Wt ☐ or Vol ☐ ?		

Test Information

Number of Samples Provided:
Tests Requested:

NASA-STD-6001	***SP-R-0022A***
☐ Test 1: Flammability: Test Atmospheres: ____% O_2 ____psia	☐ Thermal Vacuum Stability (VCM)
☐ Test 4: Wire Insulation Flammability: Test Atmospheres: ____% O_2 ____psia	☐ Special Testing (describe):
☐ Test 7: Toxic Offgassing: Flight Hardware Yes ☐ No ☐ Size: _____ Weight:	
☐ Test 13A: Ambient LOX Impact	
☐ Test 13B: High-Pressure Impact: Test Pressure: ____ psia Temperature (if GOX): ____ (°F)	
☐ Test 17: Promoted Ignition /Combustion: Test Pressure: ____ psia	
☐ Test 18: Arc Tracking: Test Atmospheres: ____% O_2 ____psia	

Notes/Remarks/Special Instructions

June 2002 **Include MSDS and Product Literature With This Worksheet** Baseline

Figure 5.
Typical Test Request Worksheet Form

Note: Representative Data Sheet. Refer to Forms Master list for current version.

Figure 6.
Typical Toxicity Log Form.

LOG SHEET

Tox Lab No.: Item No.: Material Code:

ARTICLE

Description:

Manufacturer:

Part No.: Serial No.:

Size: (L) cm in. Volume: cm^3
(W) cm in. Surface Area: $in.^2$
(THK) cm in.
Weight: g Number of Items:

Substrate: Substrate (THK) mils

Sample Preparation Cleaning No.:

TEST

Test Date: Project:

Cure No.: Priority No.:
Cure:

Composition:

Chamber: Volume: (*l*) ID No.: Free Volume: (*l*)

Requester: Test Report No.:
Company: Tracking No.:

Memo Written By: Memo No.: Date:
Remarks:

1/05 EM10-F-CHM-033

Note: Representative Data Sheet. Refer to Forms Master list for current version.

Calibration Statement: Categories IV and V Equipment

Calibration is required before use per MPR-8730.5.

(Calibration before use for each test series and periodic testing by the Using LIne Organization)

Calibration Contacts: EM10/James Perkins, EM10/Mark Griffin

User Name:_____

Equipment Description:_____

(attach multiple components sheets if necessary)

Manufacturer:_____

ECN:_____ Serial No.:_____ Model No.:_____

Date of Calibration:_____

Type of Software and Version:_____

_____

Listing of Standards Associated with Calibration:

_____

_____

_____

_____

_____

Are standards National Institute of Standards and Technology (NIST) traceable? ☐ Y ☐ N

Did calibration meet equipment manufacturer's specifications? ☐ Y ☐ N

Calibration was performed by:_____

Remarks:

_____

_____

_____

_____

_____

_____

_____

_____

1/05 EM10-F-CHM-018

Figure 7.
Typical Calibration State-

Note: Representative Data Sheet. Refer to Forms Master list for current version.

Figure 8.
Sample Rating Sheet for Assemblies Used in the Shuttle, Spacelab, and SS Airlock.

```
  MATERIAL CODE: 03587
       MATERIAL: Microgravity Plant Nutrient Experiment (MPNE)
       TEST NO.: M105426-A
         WEIGHT: 22580 g; 49.7357 lb
 CHAMBER VOLUME: 236.000 liters
         LENGTH: 8.80 in. WIDTH: 16.10 in. THICKNESS: 19.400 in.

OFFGASSED AMOUNT UNITS:  TOTAL MICROGRAMS
```

CODE	GAS NAME	MAC VALUE (mg/m^3)	OFFGASSED AMOUNT	TC
054570	2,3-DIMETHYL OXIRANE	.1	*	0
012830	2-ETHYL HEXANO	213	*	0
053700	2-PROPYL FURAN	.1	*	0
110500	ACETONE	52	103.034	3.05E-05
011600	BUTYL ALCOHOL	120.73	377.419	4.81E-05
XXX316	C5 NITROGEN COMPOUND	.1	42.384	.0063668
061800	CHLOROFORM	4.88	*	0
013600	ETHYL ALCOHOL	2000	1975.142	1.52E-05
016400	ISOPROPYL ALCOHOL	150	59.507	6.10E-06
014800	METHYL ALCOHOL	9	9944.766	.0 169996
045200	METHYL METHACRYLATE	101.88	42.576	6.43E-06
064200	METHYLENE CHLORIDE	50	550.213	.0001693
165100	OCTAMETHYLCYCLO-TETRASILOXANE	151.7	11.749	1.19E-06
035200	TOLUENE	60	*	0
065700	TRICHLOROETHYLENE	50	697.877	.0002147
168500	TRIMETHYLSILANOL	40	35.005	1.35E-05

```
* Indicates Trace Gas amounts. Traces are not used
  in calculation of T Values.

  SUMMATION OF T: .023871
 MAX LIMIT UNITS: 20.9456
          RATING: A

ACCEPTABILITY FOR ASSEMBLED ARTICLES IS DETERMINED
BY SUMMATION OF T NOT EXCEEDING 0.5
```

Toxicity Rating for Materials Used in ISS Module

MATERIAL CODE: 05582
MATERIAL: METRIGRIP 303 (.5 HR 150F CURE)
TEST NO.: M105371-A
WEIGHT: 19.41 grams; .0428 lbs
CHAMBER VOLUME: 4.23 liters
LENGTH: 7.70 in. WIDTH: 8.00 in. THICKNESS: .026 in.
OFFGASSED AMOUNT UNITS: MICROGRAMS/GRAM

CODE	GAS NAME	MAC VALUE (mg/m^3)	OFFGASSED AMOUNT	
063000	1,1-DICHLOROETHANE	100.49	*	
110500	ACETONE	52	.298	.002203
161000	CARBON MONOXIDE	10	4.438	.170641
112500	CYCLOPENTANONE	29.2	*	0
031600	ETHYLBENZENE	130	*	0
055100	GLYCOLMETHYLENE ETHER	36	*	0
016400	ISOPROPYL ALCOHOL	150	.164	.00042
035500	M-XYLENE	220	.242	.000423
014800	METHYL ALCOHOL	9	.959	.040971
063900	METHYL CHLOROFORM	163.29	*	0
035800	O-XYLENE	220	.097	.00017
140000	ORGANIC ACIDS	.1	*	0

* Indicates Trace Gas amounts. Traces are not used in calculation of T Values.

T100: .214828
MAX LIMIT WEIGHT: 232.744 lbs
RATING: K

Figure 9.
Sample Rating Sheet for Material Used in the ISS Module.

Figure 10.
Sample Preparation Data Sheet.

SAMPLE PREPARATION DATA SHEET

REQUEST #: 106729 DATE RECEIVED: 02-JUN-2000

DESCRIPTION OF MATERIAL RECEIVED:
URALANE* 5774 A/B

Lot: Batch: COLOR: BROWN

RECEIVED DATA:
WIDTH/DIAM.: N/A in. LENGTH: N/A in. THICK: N/A in. AVG. WGT.: N/A gm.

MEASURED: Y CUT: Y WEIGHTED: Y PHOTO: 6729-T

CLEAN PER: N/A

SUBSTRATE MATERIAL	THICK inches	WEIGHT grams
ALUMINUM	.00330	1.9000

SAMPLE PREP. DATA:

NHB TEST	PREPARED BY:	PREP DATE	#	WIDTH DIAM in.	LENGTH in.	WEIGHT grams
07	GARY W. GLASS	08-JUN-2000	1 D	2.27700		21.20000

CURE INFORMATION:

NR	PH	BLEND	Q	TIME hr	TEMP f	PRESS kPa
1	1	30 PARTS "A" 15 PARTS "B"	1	2	150	14.7

CURE NOTE:

DESCRIPTION OF PREPARATION METHOD:
08-JUNE-2000 TEST 07: 30 GRAMS OF PART "A" MIXED WITH 15 GRAMS OF PART "B." THE SAMPLE MATERIAL WAS PLACED IN AN ALUMINUM BOAT AND PLACED INTO A 150 DEG F PREHEATED OVEN. THE SAMPLE CURED IN THE OVEN FOR 2 HOURS. AFTE CURING THE SAMPLE WEIGHT WAS OVER THE NEEDED WEIGHT FOR A TOX TEST, SO THE SAMPLE WAS CUT SO THE WEIGHT COULD BE BROUGHT DOWN TO A TESTING WEIGHT.

8.0 Records

Records for the Building 4623 Toxicity Laboratory shall consist of (a) memoranda that contain test results and that are stored electronically in MAPTIS and (b) calibration records.

8.1 Memoranda

Memoranda containing test results shall be retained indefinitely by EM10. These memoranda shall be stored electronically in the MAPTIS database and shall be accessible by test request number or memorandum number.

8.2 Calibration Records

8.2.1. All equipment requiring calibration shall be in current calibration, in accordance with EM10-OWI-CHM-050, *Building 4623 Guidelines for General Operations.*

8.2.2. Form EM10-F-CHM-018, current revision (Figure 1, section 7.0), shall be used to document the calibration of all Category IV and Category V equipment.

8.3 Maintenance of Records

8.3.1. Memoranda less than 10 years old shall be maintained in ready-access files in MAPTIS; memoranda 10 years old or older shall be automatically transferred to historical files.

8.3.2. Calibration records shall be maintained on site for a minimum of 10 years, filed and indexed by test request number. These shall be stored in a manner that will protect them, *e.g.*, in a test folder stored in a metal file cabinet. After 10 years, calibration records shall be transferred to historical files.

8.3.3. The original test records shall be saved for a minimum of 5 years.

9.0 Tools, Equipment, and Materials

9.1 Testing Equipment

The primary instruments used in performing Test 7 are:

- Perkin-Elmer Autosystem GC/FID (Figure 11): a gas chromatograph with a flame ionization detector and a capillary RTX-1 polysiloxane column for analysis

- Finnigan INCOS XL GC/MS (Figure 12): a gas chromatograph that uses a quadrapole mass spectrometer as the detector, a capillary RTX-1 polysiloxane column for analysis, and a Tekmar purge and trap (Figure 13) for sample concentration.

- Tekmar 3100 Purge and Trap (Figure 13): a concentrator that allows automatic processing of air samples by gas chromatography.

- Finnigan Voyager GC/MS (Figure 14): a gas chromatograph that uses a quadrapole mass spectrometer as the detector, a capillary RTX-5 polysiloxane column for analysis, and a Tekmar purge and trap for sample concentration.

- Nicolet Nexus 670 FTIR: an FTIR instrument capable of scanning from 400 to 4000 cm^{-1}, equipped with a liquid nitrogen cooled mercury/cadmium/tellurium (MCT) detector and a 200-ml gas cell.

- Tekmar Aerotrap 6000 Purge and Trap: a concentrator that allows automatic processing of air samples or analysis by gas chromatography. It is equipped with a liquid nitrogen cryo-focusing accessory that allows analysis of low molecular weight organic compounds.

The GCs that are fitted with capillary columns are also equipped with the capability of cryogenic cooling to achieve optimal separation of constituents.

9.2 Required Tester Maintenance

When Toxicity Laboratory equipment requires repair or maintenance, personnel **shall notify** the Toxicity Laboratory Manager.

9.3 Calibration

Toxicity Laboratory personnel **shall perform** the following calibrations weekly and as needed:

Figure 11.
Perkin-Elmer Autosystem GC/FID for All Offgassed Constituents, with the Exception of Carbon Monoxide and Methane (capillary column).

Figure 12.
Finnigan INCOS XL GC/MS

Figure 13.
Tekmar 3100 Purge and Trap

Figure 14.
Voyager GC/MS with Tekmar Aerotrap 6000 Purge and Trap.

Note: For the mass spectrometers, a mass calibration allows the mass spectrometer to assign the correct mass values to the ion signals that it detects. The basic process of mass calibration involves the acquisition of a data file (mass spectrum) using a standard mass calibration compound. The data file is then compared with a mass calibration reference file of the same compound, which has the correct mass assigned to each peak. Any difference between these two files is adjusted to bring the new data file into line with the mass calibration file. This adjustment is then applied to all subsequent data files acquired.

9.3.1. Finnigan INCOS XL GC/MS Using Certified FC43 Gas Standard

9.3.1.1. **Start** the computer and **logon** to the network by performing the following:

- **Reboot** the acquisition computer (the machine running OS/2).
- **Double click** on "File and Print Client Workstation Logon." For the User ID, **enter** "USER;" for the Password, **enter** "PASSWORD."
- **Click** on "okay."

If the computer is already running and logged on to the network, **proceed** to step 9.3.1.2.)

9.3.1.2. Calibration Procedure: After logging on to the network, perform the following calibration steps:

9.3.1.2.1. **Double click** on the "Envirolink GC-LC/MS Program" icon.

9.3.1.2.2. **Double click** on the "Incos 50" icon.

9.3.1.2.3. **Double click** on the "Calibration" icon. The calibration screen should now be displayed. The calibration gas starts flowing automatically.

9.3.1.2.4. **Click** the F4 key after the gas has flowed about 20 seconds; this normalizes peaks.

9.3.1.2.5. **Select** "Mode;" then **select** "Tune;" then **select** "Manual."

9.3.1.2.6. **Note** the electron multiplier voltage; then **decrease** the electron multiplier voltage by selecting "EM_Voltage" and clicking on the left arrow. **Decrease** the voltage by approximately 100 volts.

9.3.1.2.7. **Click** "Close."

9.3.1.2.8. **Click** the F4 key again to normalize peaks.

9.3.1.2.9. **Select** "Mode;" then **select** "Calibrate;" then **select** "Manual." The "Mass Calibration" window should appear on screen.

9.3.1.2.10. **Select** "mass 28." A red arrow will highlight the mass approximately in the middle of the peak; *if the arrow is not very close to the peak center,* **click** the left or right arrow under "Found At" until the red arrow is as close to the center of the peak as possible.

9.3.1.2.11. **Click** "Calibrate" on the "Mass Calibration" window.

9.3.1.2.12. Perform steps 9.3.1.2.10 and 9.3.1.2.11 for the other masses (masses 69, 100, 131, 219, 414 and 502), until the red arrow is centered for each mass.

9.3.1.2.13. **Select** "mass 69"again, and then **click** on "Close" on the "Mass Calibration" window.

9.3.1.2.14. **Select** "Mode; " then **select** "Tune;" then **select** "Manual."

9.3.1.2.15. **Increase** the "EM_Voltage" to the same setting it was at the beginning of this procedure (see step 9.3.1.2.6); then **select** "Close."

9.3.1.2.16. **Wait** about 10 seconds, then **click** the F4 key again to normalize peaks.

9.3.1.2.17. **Select** "File;" then **select** "Save."

9.3.1.2.18. **Select** "File;" then **select** "Print Report."

9.3.1.2.19. **Exit** the calibration screen by selecting "File" and then selecting "Exit."

9.3.1.2.20. **Place** the results and a completed EM10-F-CHM-018 form in the notebook labeled "Calibration for Finnigan GC/MS INCOS50."

Note 1: An internal calibration gas, FC-43, is used for the calibration.

Note 2: This internal calibration shall be performed before each day of toxicity testing

Note 3: Calibration of the instrument using FC-43 involves comparison of acquired data to the calibration reference table. This table lists the exact masses and relative abundance of the major ions in the FC-43 mass spectrum. **Compare** the results obtained with previous data obtained at the same electron multiplier voltage. *If the results are acceptable,* **commence** with sample analysis.

Note 4: After the calibration is complete, the report printed shall be saved for record in the Toxicity Laboratory for a minimum of 10 years.

Note 5: Refer to the Finnigan INCOS Series GC/MS Systems Getting Started Manual, the INCOS XL Series Systems Operator's Manual (Vols. 1&2), and the Tekmar LSC 3100 User's Manual for additional information about these systems.

Note 6: Refer to the Envirolink GC-LC/MS Users Manual for detailed calibration information.

9.3.2. Voyager GC/MS Using Certified FC43 Calibration Gas Standard

9.3.2.1. From the main Windows screen, **select** "Start;" then **select** "Programs;" then **select** "Xcalibur;" and finally **select** "Xcalibur" again.

9.3.2.2. **Select** "Instrument Setup."

9.3.2.3. **Select** "File;" then **select** "Open;" then **double click** on "Toxlab."

9.3.2.4. **Click** on the Voyager image; **select** "Analysis;" then **select** "Tune."

9.3.2.5. **Click** on both the "toggle operate" and "toggle gas" icons to switch them on.

9.3.2.6. **Wait** about 15 seconds, and then **print** this view to the HP1220 printer. The Mass Spectrometer settings and the major mass intensities will print.

9.3.2.7. **Click** on the "toggle operate" and "toggle gas" to turn them off.

9.3.2.8. **Select** "View;" then **select** "Calibrate."

9.3.2.9. **Select** "Calibration;" **select** "Calibrate;" then **select** "Start" in the "Instrument Diagnostics and Optimizations" window.

9.3.2.10. **Select** "Start." An internal calibration is automatically performed, using the FC43 calibration gas standard. The calibration takes 3 to 4 minutes.

9.3.2.11. When the calibration is completed, the calibrate box contains a green check mark. The standard deviation (SD) should be 0.05 or less. *If the calibration is successful,* **select** "Close." **Proceed** with step 9.3.2.12.

If the calibrate box contains an X mark, **perform** the following:

9.3.2.11.1. *If the SD is greater than 0.05,* **run** the calibration again (steps 9.3.2.9 through 9.3.2.11.)

9.3.2.11.2. *If, after 2 calibration attempts, the SD is still greater than 0.05,* either **perform** an Autotune (on the "Instrument Diagnostics and Op-

timizations" window) or s**lowly increase and decrease** different tune parameters on the Tune Screen until better peaks are observed. **Rerun** the calibration after trying these solutions.

9.3.2.12. **Select** "File;" **select** "Save As;" then **select** "Calibration Settings." **Save** the file as the file name "Voyager."

9.3.2.13. **Print** the calibration to the HP 1220 printer.

9.3.3. Finnigan INCOS XL GC/MS Calibration Check Using Alkane Standard

Analyze a certified Alkane Standard to ensure that the individual constituents are detected, that the proper elution times through the column are maintained, and that the proper masses are present to identify the standard constituents.

An analysis of a Certified Gas Standard containing straight chain alkanes shall be performed by the following procedure, which also serves as the guide for toxicity sample data acquisition:

9.3.3.1. **Run** the FC43 gas calibration procedure as described in Finnigan INCOS50 GC/MS Calibration. **Verify** the data are acceptable.

9.3.3.2. **Load** the "Toxlab" method:
- **Double click** on "Acquisition" icon.
- **Select** "Window;" then **select** "Method."
- **Select** "File;" then **select** "Open."
- **Double click** on TOXLAB.MTH.

9.3.3.3. **Select** "Edit", then **select** "Parameters."

9.3.3.4. **Enter** the pertinent sample and file information. The file name will be the next number in the series, *i.e.,* alkane22, alkane23. **Click** "**Okay.**"

9.3.3.5. **Select** "Acquire;" **select** "Download;" then **select** "Start.

9.3.3.6. **Press** "Start" on the Tekmar 3100 Purge & Trap remote keyboard. The Tekmar 3100 goes into "Purge" mode for 1 minute 15 seconds to allow time for sample injection.

9.3.3.7. **Inject** 5 cc of the alkane gas standard into the Tekmar Purge and Trap System . **Verify** that the expiration date on each container of standard has not been exceeded.

9.3.3.8. The Tekmar then desorbs the gas standard onto the column of the Finnigan GC/MS.

9.3.3.9. The calibration standard is analyzed using the same time/temperature programs as the Toxicity Analysis Method (TOXLAB.MTH).

9.3.3.10. **Verify** the identification of each compound with multiple library searches (toxlab and NIST libraries). All peaks are identified by the Mass Spectrometer using the fragmentation pattern of each compound.

9.3.3.11. After the analysis is completed, **identify** each compound, and **verify** the elution times.

9.3.3.12. **Double-click t**he "Data Analysis" icon.

9.3.3.13. **Select** "Chromatogram" and then "Extract Ion Chromatograms."

9.3.3.14. In the upper window, **add** the Time Range "3.0 to 12.0 minutes."

9.3.3.15. In the lower window, **add** the Ions "29" in number one and "43" in number two.

9.3.3.16. **Print** the window displayed by selection "File," then "Print," and then "TIC and Spectrum."

9.3.3.17. **Verify** that the intensities are similar to the previous results. (**See** the notebook titled *Calibration Alkane Std*.) **Place** the chromatogram in this same notebook.

Note 1: The chromatogram is printed and kept for record a minimum of 10 years.

Note 2: This procedure uses the same alkane standard as that used for the Autosystem GC.

Note 3: Refer to the Finnigan INCOS Series GC/MS Systems Getting Started Manual, the INCOS XL Series Systems Operator's Manual (Vols. 1&2), and the Tekmar LSC 3100 User's Manual for additional information about these systems.

Note 4: This calibration is performed at least monthly or as needed*, i.e.,* following instrument maintenance and repair.

Note 5: NASA-STD-6001 states that the Gas Standards (Mixtures A and B) **shall be analyzed** every 3 months. The measured concentrations **shall** be within 25% of the specified concentrations.

9.3.4. Perkin-Elmer Autosystem GC/FID (capillary column)

Analyze a certified Alkane Standard to ensure that all the constituents are detected while correct elution times and area counts are maintained. A complete analysis using a certified gas standard is performed by the following procedure:

- Inject 1 cc of the gas standard onto the column. (The standard contains known straight chain hydrocarbons in known amounts)
- The calibration standard is analyzed using identical time/temperature programs as the toxicity analysis.
- The standard is analyzed each day of toxicity testing with a chromatogram kept for record.
- The chromatogram is used to verify all peaks have correct elution times and area counts.
- The area counts of the propane (retention time approximately 1.5 minutes) for the two runs are entered in the MAPTIS database (see section 4.7.1) for use in the toxicity calculation.
- After all verifications, the instrument is ready for use.
- Refer to the Perkin Elmer Operator's Manual for the GC and PE Nelson Model 1022 Personal Integrator Installation and Setup Guide.

Note: NASA-STD-6001 states that the Gas Standard (Mixtures A and B) **shall** be analyzed every 3 months. The measured concentrations must be within 25% of the specified concentrations.

9.3.5. Agilent 6890 GC/FID (packed column) for Carbon Monoxide and Methane

The procedure for this calibration is the same as for the Autosystem GC/FID (section 9.3.4), using certified gas standards. **Perform** analyses with certified standards of CO and CH_4 to ensure proper elution times and that area counts are maintained. **Refer** to the *Agilent 6890 Series Gas Chromatograph Operating Manual* and the *Understanding Your ChemStation* manual.

9.3.6. Nicolet Nexus 670 FTIR Calibration (using formaldehyde and ammonia gas standards)

9.3.6.1. **Verify** that the 2-meter gas cell is in place in the Nexus 670 FTIR in the sample accessory compartment. *If the cell is not in place,* **set** it in the accessory compartment so the pin in the front and the rear of the gas cell line up with the pins in the FTIR.

9.3.6.2. **Tighten** the collars on either side of the gas cell so they are firmly against the body of the FTIR on either side of the gas cell.

9.3.6.3. **Double click** on the Omnic icon on the computer desktop.

9.3.6.4. **Select** "Collect," then select "Experiment Setup".

9.3.6.5. **Select** "open", then d**ouble click** on the following file: G:\omnic 6.0\My Documents\spectra\Param\exp 2M gas cell.

9.3.6.6. **Verify** that the polyethylene tubing leading from the wall-mounted gauge is connected to one of the gas cell valves.

9.3.6.7. The valve referenced in step 9.3.6.6 shall be fully open (counterclockwise). The other valve on the gas cell shall be fully closed (clockwise). The valve above the vacuum pump shall be fully closed (clockwise).

9.3.6.8. **Plug** in the vacuum pump to turn it on.

9.3.6.9. Fully **open** the valve above the vacuum pump (counterclockwise). This causes wall-mounted gauge reading to start dropping immediately and the pressure to drop to 0.1 to 0.2 psi.

9.3.6.10. In the Omnic software, **select** "Collect," and then **select** "Collect Background." **Click** "Okay" to begin data collection. **Allow** the background run to finish (100 scans). For reference, the background spectral signal increases in intensity from 4000 to 1500 cm^{-1}, then decreases from 1500 to 400 cm^{-1}. *If the background looks at all irregular (excessive noise, etc.),* it may be necessary to let the vacuum pump run a few minutes longer to remove all the CO_2 and water. The spectra displays as the background is running.

9.3.6.11.. When the window appears after data collection asking whether to display the background spectra in the main window, **select** "No."

9.3.6.12.. **Close** the valve above the vacuum pump.

9.3.6.13. **Open** the stem valve of the formaldehyde standard bottle (counterclockwise).

9.3.6.14. **Turn on** the regulator by opening it one full turn (clockwise).

9.3.6.15. **Note** the pressure on the wall-mounted pressure gauge. (The pressure should be 0.1 to 0.2 psi). *If the pressure is not 0.1 to 0.2 psi,* **verify** proper vacuum pump operation, and **pump** down the gas cell again.

9.3.6.16 **Slowly open** the hand valve on the formaldehyde standard slightly; the pressure on the wall-mounted gauge should increase by 0.2 to 0.3 psi. *If the pressure increases too much,* **vacuum** the cell down again and **add** 0.2 to 0.3 psi of the formaldehyde standard.

9.3.6.17. **Close** the stem valve, regulator and hand valve on the formaldehyde standard bottle.

9.3.6.18. In the Omnic software, **select** "Collect Sample," then **select** "Okay" to begin data collection.

9.3.6.19. When the window appears asking whether to display the sample spectra, **select** "Yes."

9.3.6.20. **Save** the standard in the folder G:\omnic 6.0\My Documents\spectra\Param\tox samples.

9.3.6.21. **Look** for peaks at 2897 cm^{-1} and 1745 cm^{-1} of the spectra that indicate the formaldehyde. *If there is no peak at 2897 cm^{-1}* (it will be about 2 or 3 times the height of the baseline itself), it may be necessary to add a little more of the standard. *If this happens*, **repeat** steps 9.3.6.15 through 9.3.6.20 above and add 0.2 to 0.3 psi. of formaldehyde standard.

9.3.6.22. **Repeat** steps 9.3.6.9 through 9.3.6.20 above for calibration of the FTIR for ammonia, using the ammonia standard bottle rather than the formaldehyde standard bottle. **Open** the stem valve of the ammonia standard, and **open** the ammonia regulator until the pressure of the regulator reads 20 psi. **Increase** the pressure on the wall-mounted pressure gauge to 0.5 psi. The peaks for ammonia are at 931 cm^{-1} and 966 cm^{-1}.

CAUTION: Do not allow the pressure of the gas cell to ever go above 25 psi. *If it reaches this pressure,* the gas cell window may burst. If the pressure raises above 25 psi, **pull** a vacuum on the gas cell IMMEDIATELY.

9.3.7. **Complete** a calibration data sheet (see Figure 7 for a sample of form EM10-F-CHM-018) after each of the above instrument calibrations. **Print** a copy of each of the standards spectra.

9.3.8. **Place** the calibration data sheets and spectra in the FTIR Calibration Standards Notebook.

9.4 Toxicity Laboratory Equipment

Table 1 contains the Toxicity Laboratory Equipment List. Table 2 lists the toxicity chambers and their volumes.

Table 1.
Toxicity Laboratory Equipment List

	NEMS or ID #	Location	Room
Mass Spectrometer Equipment			
Finnigan Incos 50 XL GC/Mass Spectrometer	1281763	Bldg. 4623	102
Finnigan Voyager GC/Mass Spectrometer	1937593	Bldg. 4623	102
Tekmar 3100 Purge and Trap	2014752	Bldg. 4623	102
Frontier Computer	2012074	Bldg. 4623	102
Xerox Docuprint 2825 printer	2015184	Bldg. 4623	102
Dell Optiplex GX 260	000612785	Bldg. 4623	102
Dell 17” monitor	OAO 521465	Bldg. 4623	102
Hitachi 21” monitor	1893076	Bldg. 4623	102
Dell 19” monitor	1963424	Bldg. 4623	102
Prolab Data Acquisition System	2012073	Bldg. 4623	102
Cylinder of Helium	NA	Bldg. 4623	102
LN2 Dewar	NA	Bldg. 4623	102
HP Deskjet	1220C	Bldg. 4623	102
19” Monitor	X-970	Bldg. 4623	102
Tekmar Aerotrap 6000	USO3120001	Bldg. 4623	102
Gas Chromatograph Equipment			
PE Nelson 1022 Integrator	1396454	Bldg. 4623	102
Agilent 6890 GC	2016125	Bldg. 4623	102
Agilent 6890 GC	2016126	Bldg. 4623	102
HP Kayak DT XM600 computer	2016158	Bldg. 4623	102
Perkin-Elmer Autosystem GC	1219430	Bldg. 4623	102
Compaq 21” monitor	OAO 501708	Bldg. 4623	102
Cylinder of Helium	NA	Bldg. 4623	102
LN2 Dewar	NA	Bldg. 4623	102
Cylinder of Hydrogen	NA	Bldg. 4623	102
HP Laserjet	4100	Bldg. 4623	102
Miscellaneous Equipment			
Laminar flow bench	G031302	Bldg. 4623	100B
Mettler PJ600 Balance	0065194	Bldg. 4623	100B
Blue-M Oven	1965473	Bldg. 4623	100B
Grieve, Lab Oven	G030359	Bldg. 4623	100B
Trivac D4A vacuum pump	1443869	Bldg. 4623	102
AND FW150K Floor Balance	M621162	Bldg. 4623	102
Digital LN17 printer	1896537	Bldg. 4623	100A
HP Laserjet 6P printer	1896631	Bldg. 4623	100A
Precision Sampling Co. 1cc and 10cc syringes	NA	Bldg. 4623	102
Tool Kit	TC-100/ST	Bldg. 4623	102
Tool Kit	TC-150/ST	Bldg. 4623	102
Dandy Pallet Lift	1401394	Bldg. 4623	100B
2-Stage Regulators	18A/18B	Bldg. 4623	102
Simco Aerostat Ionizers	M637015	Bldg. 4623	100B
Simco Aerostat Ionizers	M637016	Bldg. 4623	100B
Westronics Strip Chart Recorder Series 2600	1740912	Bldg. 4623	100B
Westronics Strip Chart Recorder Series 2600	1740911	Bldg. 4623	100B
Cole-Parmer Digital Thermometer	M635051	Bldg. 4623	102
Cole-Parmer Digital Thermometer	M635052	Bldg. 4623	102
Varian vacuum pump	SD450	Bldg. 4623	102

CHAMBER VOLUME (liters)	DIMENSIONS (in.)	QUANTITY
.99	4.0 (dia.) x 4.8 (l)	2
1.00	3.7 (dia.) x 5.9 (l)	3
2.16	3.9 (dia.) x 11.8 (l)	1
2.19	4.0 (dia.) x 11.875 (l)	3
4.23	6.3 (dia.) x 8.0 (l)	17
12.45	11.8 x 7.5 x 7.5	1
22.42	21.5 x 7.8 x 7.8	1
28.09	12.0 x 12.0 x 12.0	1
34.75	16.0 x 10.8 x 12.3	1
45.37	16.0 x 16.0 x 11.0	1
236.86	36.25 x 16.5 x 24.25	2
537.086	40 x 43.9 x 26	1
1555.2	46.8 x 43.9 x 43.9	1
8155.2	46.0 x 71.0 x 143.5	1

Table 2.
Toxicity Chambers and Volumes.

10.0 Personnel Training

The nature of testing that occurs in the Building 4623 Toxicity Laboratory is complex and involves potential hazards; therefore, all toxicity testing shall be performed only by credentialled Toxicity Laboratory personnel or under the direct supervision of personnel credentialled to perform Test 7.

10.1 Credentialling

Candidates for credentially in support of Test 7 shall have:

- A B.S. in chemistry or a technical degree with equivalent experience
- Formal training by an instrument's manufacturer or other qualified personnel
- Electrostatic discharge certification (required for handling flight hardware)
- Training in the following subjects:
  - High-pressure systems (>150 psig)
  - General safe laboratory practices
  - Hazardous waste disposal
  - Cryogen handler
  - Hydrogen handler/user
  - Inert/asphyxiant gases and liquids

To complete credentially, the candidate **shall**:

- **Read** the OWI thoroughly, and sign a statement of reading and understanding the OWI. Each candidate shall be issued a personal copy of the OWI.
- **Complete** a minimum of 60 hours of hands-on testing with another operator who is credentialled in Toxicity Testing. As a minimum, this training **shall include:**
  - Familiarization with all laboratory instrumentation operating procedures
  - Toxicity sample loading and log book entry procedures
  - Search and identification of mass spectra
  - Log book data entry and MAPTIS data entry procedures.
- **Demonstrate** knowledge of the test and Toxicity Laboratory equipment by completing two successful test sets under the supervision of the test engineer. The supervisor **shall provide** certification documentation.
- **Pass** a written test covering the OWI and administered by the test engineer.

Records **shall be kept** on file as proof of training. These records **shall include** training expiration dates and required refresher courses. Verification of credentials **shall consist** of a copy of the written test, the signed statement, and the training record. The contract project manager **shall inspect** these records and **shall issue** a credentials card.

10.2 Recredentialling

Recredentialling **shall be** automatic if there has been no break in service over 6 months and the candidate has continuously performed duties in the credentialed area. Otherwise, recredentialling **shall require** completion of the credentially steps above.

EMERGENCY PHONE NUMBERS

Emergency..... 911

Medical Center..... 4-2390

Industrial Safety..... 4-0046

Chemical Spills..... 4-4357

Safety Monitor
Building 4623..... 5-0358